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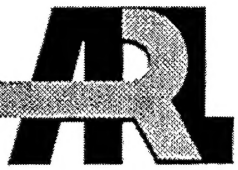

Surface Phenomena of Solid Propellants Ignited by Plasma

by Pamela J. Kaste, Amy E. Kinkennon, Robert J. Lieb, Avi Birk,
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ARL-TR-2500 May 2001

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Abstract

The morphological and chemical characterization of M30 propellants recovered after conventional and plasma ignition with polyethylene (PE) terephthalate (Mylar) in an interrupted closed bomb (extinguished at pressures between 35 and 100 Mpa) has been performed. It has been reported that burning rate augmentation appears to occur during the plasma event, but there was no evidence for post-plasma augmentation. For the extinguished grains from PE plasma ignition, there appears to be removal of nitroguanidine (NQ) crystals in the perforations. Also, hot embedded particles and increased surface area were observed, instead of the melt layer normally obtained with conventionally ignited samples. Although extensive chemical characterization has been performed, there appears to be very little chemical difference between the burned surfaces and subsurfaces of the plasma and conventionally ignited samples. Scanning electron microscopy (SEM) analysis revealed that although a melt layer is present on the surface of M30, it is immeasurably thin. Moreover, the SEM analysis showed that NQ depletion in the perforations was most evident at the lowest blow-out pressure used (35 Mpa) and seemed to diminish at higher pressure, apparently becoming obscured by "normal" burning processes. Thus, plasma-propellant interactions and associated effects may occur only very early during ignition, and it may be that there are few decomposition products remaining near the surfaces of the samples.

Acknowledgments

Dr. D. Kooker of the U.S. Army Research Laboratory (ARL), Aberdeen Proving Ground (APG), MD, spent much time in helpful discussions, and his technical insights are very much appreciated. Dr. Mike McQuaid of ARL, APG is also thanked for his consultations in the area of capillary physics.

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1. Introduction

The interaction of plasmas with solid propellants is being investigated using an interrupted closed chamber capable of either plasma or conventional black powder ignition. The ultimate goal is to be able to use plasma ignition to tailor the burning characteristics of solid gun propellants so that the temperature sensitivity in gun systems is minimized. In previous, noninterrupted closed-bomb analyses, the effect of plasma ignition on propellant burning rate was investigated. It was reported [1] that for M30, a 33% increase in burning rate over the range of 100–220 MPa occurred with plasma ignition, relative to conventional ignition. The plasma augmentation of JA2 burning rate was insignificant. Researchers in the international community have also reported augmentation of the burning rate with nitramine-based composite propellants [2]. In the initial noninterrupted burn (studies with M30), the plasma was generated by ablating a polyethylene (PE) capillary [1].

Further research [3] included interrupted burn experiments with both conventional ignition and plasma ignition using PE capillaries for the dual purpose of (1) determining whether the increases in burning rate for M30 were intrinsic to the propellant or the result of an increase in surface area due to fracture [4] or porous burning, and (2) characterizing extinguished grains to understand the ignition and combustion chemistry that occur with the two igniters. The approach was to repeat key experiments from Del Güercio [1] with M30 and JA2 propellant, but interrupt the burning at 35, 75, and 100 MPa and collect the extinguished propellant for physical examination and chemical analysis. The interrupted closed-bomb experiment offers a means of studying basic plasma-propellant interactions under well-defined conditions of plasma energy and power input, pressure profile, and proximity of the sample to the plasma. Since the blowout pressure can be varied, this can be done as a function of very early ignition (plasma interaction with propellant dominant) and combustion (burning of propellant dominant). Characterization of residual propellant from both plasma and conventionally ignited samples provides a method for studying the ignition and combustion chemistry that occur with the two igniters.

In a previous study [3], it was not possible to conclude whether or not plasma augmentation of the intrinsic propellant burning rate occurred. The fact that the pressure-time (P-t) curves of the plasma-ignited grains show an initial rapid pressurization, due mainly to plasma injection rather than propellant burning, made it impossible to compute the propellant burning rate using the P-t curve and measured regression of the extinguished grains using BRLCB [5] from a single trial. Overlapping P-t traces from trials at different blow-out pressures, for which the initial pressurization was comparable and would cancel if differences in grain regression were considered, were required. Unfortunately, plasma ignition using PE capillaries made it difficult to achieve reproducible P-t traces [6]. Moreover, even the extent of grain fracture with plasma vs. conventional ignition was not definitive due in large part to the variability in plasma parameters [3].

To improve the reproducibility of the plasma ignition parameters so that plasma burning rate could be assessed and the effect on plasma-propellant interactions of alternative chemistries could be studied, other capillary materials were used [6, 7]. Polyethylene terephthalate (Mylar) capillaries met the criteria of prompt pressure rise and high-power input to the chamber, while preventing premature breakdown of the pulse, prior to the desired 1.2-ms duration. Mylar also allows investigation of CHO chemistry, rather than the more simple CH chemistry of PE. A conclusion from the previous study was the need to rapidly recover and sample the surface of extinguished grains, based on the difficulty of isolating decomposition products from the partially burned grains [3]. Only the very outer surfaces (i.e., less than 50 μm) of the recovered grains showed any evidence (e.g., visual inspection, or chemical, morphological analysis) of decomposition, and no subsurface reaction was detected [8, 9]. Plasticizer migration from the bulk of the grains to the outside occurs within hours and could lower detection limits for decomposition products [10]. Thus, in this work, outer surfaces were promptly sampled. Morphological analyses and chemical characterization of extinguished propellant grains from the interrupted burn experiments using Mylar-based capillary igniters has been performed. The results are compared with those obtained using PE-based capillaries and are the subject of this report. Since the previous work was presented, the use of Mylar capillaries enabled an

assessment of plasma augmentation to be made. Details of those results are reported by Birk et al. [7] and Kooker [11].

2. Experimental

2.1 Closed-Bomb Firings. The samples and interrupted closed-bomb configuration are similar to those described previously [3], with the exception that, for some plasma firings, Mylar was used as the capillary material. The most important details are provided for convenience. Samples for closed-bomb analysis consisted of 7-perforated grains of M30 and JA2, with a diameter, length, and perforation diameter of approximately 0.75, 1.5, and 0.07 cm, respectively. Closed-bomb analyses of M30 and JA2 were performed in a 3.81-cm inside diameter (ID) closed bomb of 129-cm^3 volume, with a typical propellant charge weight of 32 g and with both conventional and plasma ignition. For extinguished propellant measurements, the addition of the interface of the bomb to the evacuation chamber yields a total closed-bomb area of 150 cm^3 (Figure 1). The expansion chamber consists of a 240-liter tank, with a 2.5-cm-diameter blowout area interfaced to the closed bomb. Rapid extinguishment of the propellants occurs due to the sudden expansion into the evacuated tank. Soft capture of the propellant is achieved with a lining of thermally resistant polyurethane foam. In the case of plasma ignition, the plasma is channeled through a straw that is perforated to resemble a piccolo tube, with 24 holes to enable uniform distribution of the plasma around the propellant (Figure 2). In the conventional mode, an electric match is used to ignite 0.6 g of black powder confined in a plastic straw, with the propellant distributed concentrically in two tiers around the straw (Figure 3). The plasma is generated by an electrical pulse to a nickel fuse wire, which is rapidly vaporized. This results in ionization of the PE liner, and a high-current discharge is sustained. The time interval for injection is about 1.2 ms. Closed-bomb trials for which the pulse length was less than 0.9 ms were rejected. The electrical pulse is generated with a 400-kJ capacitor-based pulse-forming network, with a charging voltage of 4 kV and an output energy of up to 29.3 kJ.

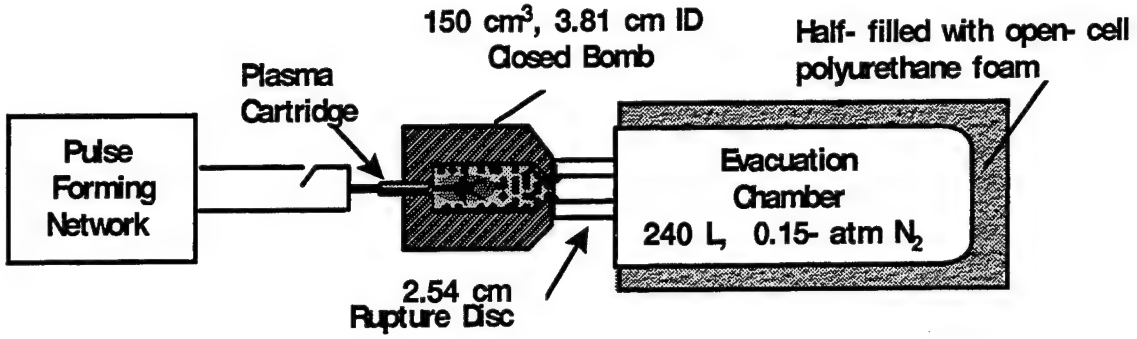


Figure 1. Apparatus for Extinguished Closed-Bomb Experiments With Propellant Soft Capture.

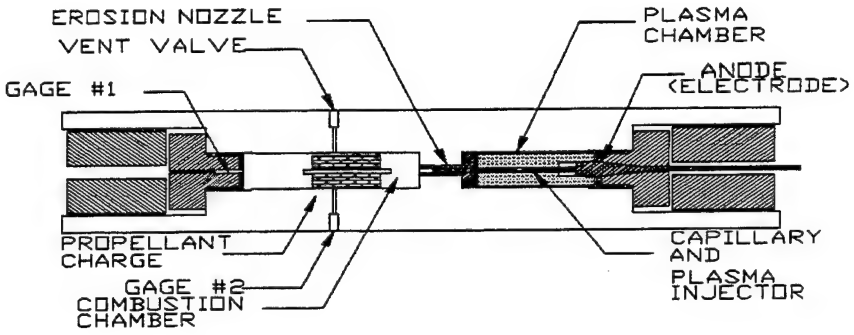


Figure 2. Schematic of the Plasma Igniter.

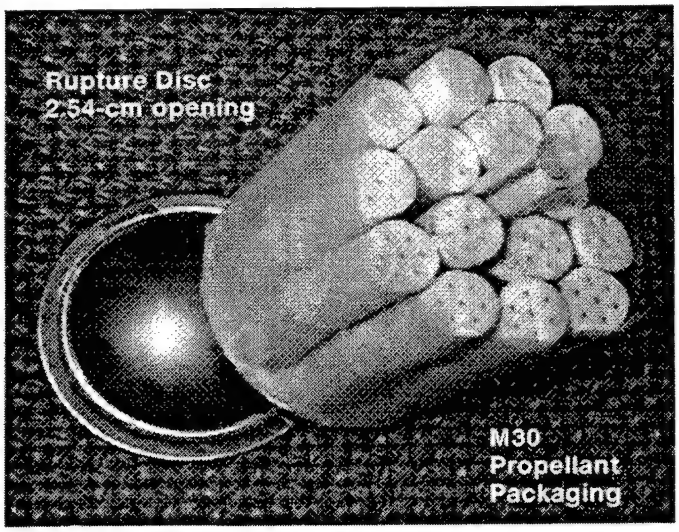


Figure 3. Packaging of M30 Propellant.

2.2 Grain Preparation and Archaeology. It was desirable to track the original location of the recovered grains to assess whether this might affect such factors as the quantity or location of residue deposited on the grains, level of decomposition or extent of burning of the grains' surface, or any morphological features observed. A simple scoring scheme was devised, which would indicate the following: (1) whether the grain was in the top or bottom tier (the bottom tier is defined as that closest to the igniter); (2) whether the grain was located in the inner or outer ring around the straw, which simulated the piccolo igniter; (3) the end of the grain that faced downward (i.e., toward the igniter); and (4) which part of the grain faced radially outward (i.e., away from the igniter). This was accomplished, as shown in Figure 4, that shows the appearance of a top tier and bottom grain as viewed end-on from the surface that would be closest to the blow-out disc. A single score was indicative of top-tiered grains; a score through one perforation was used to mark grains adjacent to the straw (inner ring, typically 5 grains), and a score through two perforations indicated that the grain was located in the outer ring (typically 10 grains). Two scores indicated that the grains were located in the bottom tier (closest to the igniter). Scores along perforations on consecutive radii indicated that the grains were in the outer packing ring, while those grains with two scores along alternate radii indicate that the grain was packaged adjacent to the straw. The grains were positioned so that the scoring pointed radially outward. Scoring was achieved with a razor and was sufficiently deep so as to be evident in the recovered grains but did not induce fracture or significantly increase surface area.

After firing, grains were separated according to where they were recovered: (1) remaining in the closed bomb, (2) loose in the 240-liter chamber in which the grains were extinguished, or (3) embedded in the polyurethane foam used to line the extinguishing chamber. For each trial, a matrix was established in which the number of grains from each original and final position was recorded. In the previous series of extinguished closed-bomb runs in which the PE capillaries were used, it would have been helpful in some of the interpretation of the scanning electron microscopy (SEM) to have known the original and final positions of the grains. Thus, the effort required to track the grains seemed a worthy investment for the series based on the Mylar capillaries.

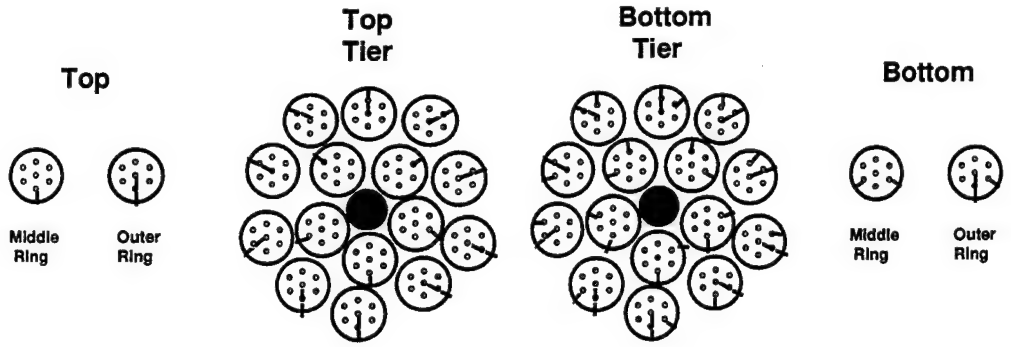


Figure 4. Scoring Scheme Used to Identify the Packaging Position and Orientation of the Grains Prior to Closed-Bomb Analysis.

2.3 Liquid Chromatography - Mass Spectroscopy (LC-MS). LC-MS was used to investigate decomposition or depletion of the components of JA2 and M30. An HP 1090 liquid chromatograph was used with a 59980B LC-MS interface and 5989B mass spectrometer. In the case of JA2, the components analyzed were nitroglycerin (NG), diethylene glycol dinitrate (DEGDN), and Akardite II. NG and nitroquanidine (NQ) were determined for M30 propellants. Outer shavings of the propellant grains were extracted overnight. Extraction solvents were ether (for JA2) and 2:1 methanol:water (for M30); both solvent systems were chosen to exclude nitrocellulose (NC) in the extract. The ether was evaporated, and the JA2 extract was redissolved in 2:1 methanol:water. Samples were passed through cellulose filters (0.2 μm) prior to injection of 25-ul samples. A C18 microbore column (100 × 2.1 mm; 5-μm bead size) was used in the separation. The elution solvent was 50:50 MeOH:water at 0.3 ml/min. The mass spectrometer was run in the electronic ionization mode; ions between 34 and 200 m/z ratio were monitored.

2.4 Scanning Electron Microscopy (SEM). Micrographs of virgin and extinguished grains were examined to establish any morphological differences between grains ignited by conventional and plasma sources. The grains were selected and cold-fractured along the longitudinal grain axis to expose the burning surface of the perforations and a cross section of the burning surface and the propellant below this surface. The prepared specimens were sputter coated with gold palladium. The lateral exterior burn surface was also examined for any differences.

2.5 Fourier-Transform Infrared (FTIR) Analysis. Microreflectance FTIR was used to obtain spectra of the propellant surface (i.e., about top 10 μ) with no modification of the sample. FTIR analyses were performed using a Mattson Polaris spectrometer operating at a resolution of 4 cm^{-1}. The reflectance spectra were obtained using a Spectra-Tech (Shelton, CT) microreflectance attachment with 32× infrared (IR) objective and signal averaging 200 scans. Aluminum foil was used to collect the background spectra. Samples for microreflectance must be flat, since curvature may distort the focus. M30 samples were prepared by microtoming.

2.6 X-ray Fluorescence (XRF) Spectroscopy. XRF spectroscopy was performed on the extinguished grains in order to identify metals present in the plasma that may be incident on the propellant samples. Before obtaining XRF spectra, the propellant samples were coated with carbon using a carbon-coating instrument (SPI Supplies Inc, West Chester, PA). The propellant-to-carbon fiber distance was greater than 3.5 cm, and a minimum voltage was used to minimize exposure of the sample to heat. Propellant grains were cooled to dry-ice temperature and split with a knife blade. The knife blade was laid vertically against the propellant grain and struck with a hammer, and a clean split was obtained. Spectra were obtained using a Kevex model 3600-0374 XRF detector interfaced to a Kevex Delta Class Analyzer with Kevex Quantex software, version V; this was interfaced to a JEOL 820, scanning electron microscope.

3. Results

3.1 Closed-Bomb Firings. The apparent plasma burning-rate augmentation of M30 was greater for the PE capillary, as compared to the Mylar capillary (Figure 5). Because of the limitation of total number of firings that could be performed and the more reliable and reproducible results using the Mylar capillary, it was decided that Mylar was preferable to PE, in spite of the lower apparent burning-rate augmentation.

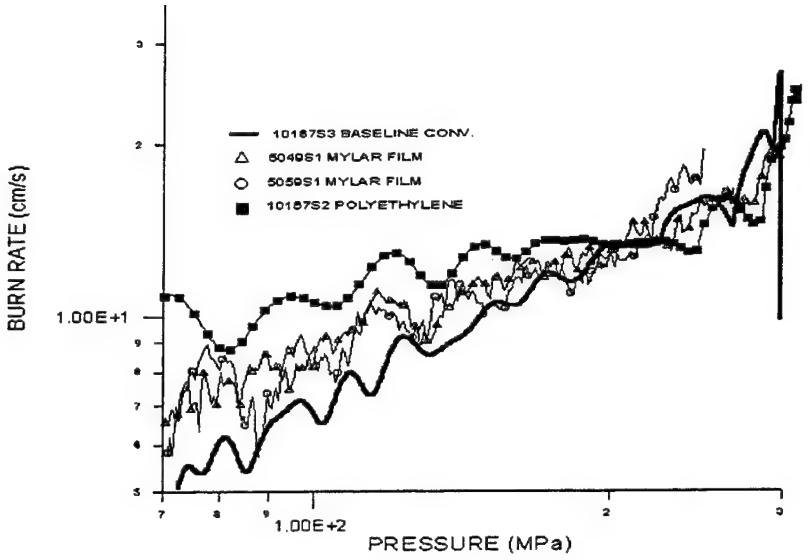


Figure 5. Apparent Burning Rates of M30 Ignited With Mylar- and PE-Based Plasmas in Noninterrupted Closed-Bomb Trials.

The power curve was also considered when evaluating each trial with plasma ignition. Figure 6 shows two plots for which the initial rise is similar, but the area under the curves is much different, yielding input energies of about 19 for the M30 samples and 29 kJ for the JA2 samples. The initial rise is comparable for all samples so that the strain rates applied to the propellants would be expected to be comparable. Excessive strain rates that might cause fracture as an artifact were thereby avoided.

The P-t curves for the M30 and JA2 samples ignited by plasma are shown in Figure 7, for blow-out pressures of nominally 35 and 100 MPa. P-t curves for conventionally ignited M30 are also shown and are evident from the longer time to burst of about 10 ms at 60 MPa. Also apparent is the uniform curvature due to the regular gas generation of the conventionally ignited propellant. In contrast, propellants ignited by plasma show a sharp increase in pressure within the first 1–2 ms due to the sudden plasma impulse, followed by a lower rate of pressure increase when normal propellant burning takes over. In the case of JA2, early experiments with the PE capillaries were used for plasma generation, and curves with overlying traces for the initial pressure rise were obtained. Overlapping curves for M30 Mylar capillaries were used for the

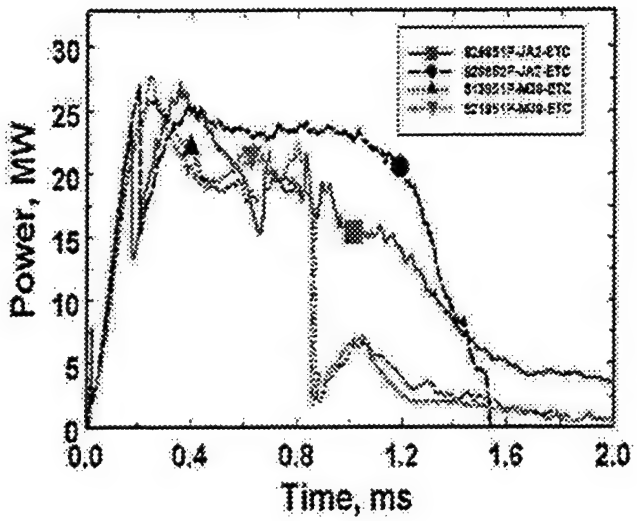


Figure 6. Electrical Power Curve for Two Plasma Ignition Trials Yielding Different Input Energies (18.4 and 29.3 kJ).

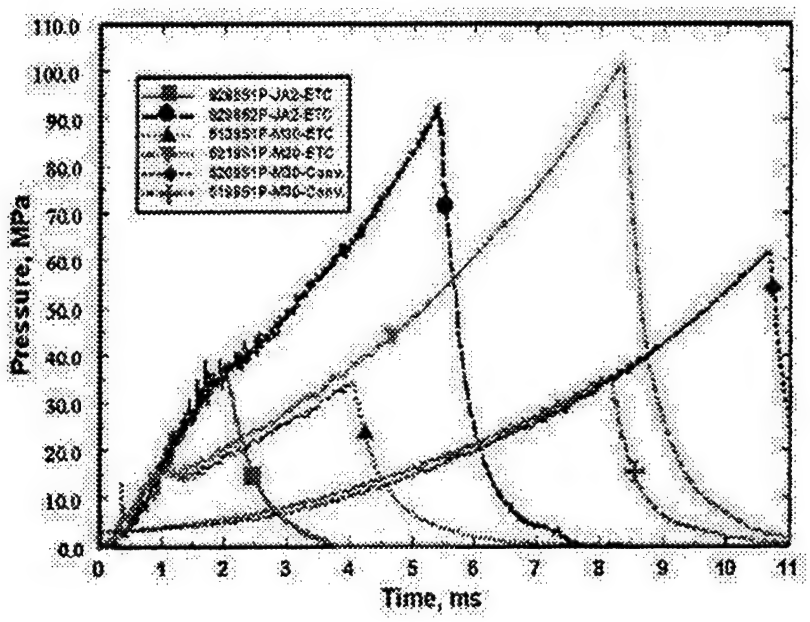


Figure 7. P-t Curves for Plasma-Ignited M30 (Mylar Capillary) and JA2 (PE Capillary) Firings, Along With Conventional Firings for M30, for Interrupted Closed-Bomb Trials.

M30 series. As seen in Figure 7, the PE capillary yields a greater initial pressure increase than Mylar if conditions of input energy and power pulse duration are comparable. This is consistent with empty-chamber, closed-bomb experiments for which the pressure rise with PE capillaries was greater by about a factor of 2 over that of Mylar during the first 2 ms [6]. Although the propellants are also different for these two scenarios, the differences in burning rate of JA2 and M30 would not account for the differences in pressure rise seen for the M30 and JA2 electrothermal chemical (ETC) curves between 1 and 2 ms in Figure 6.

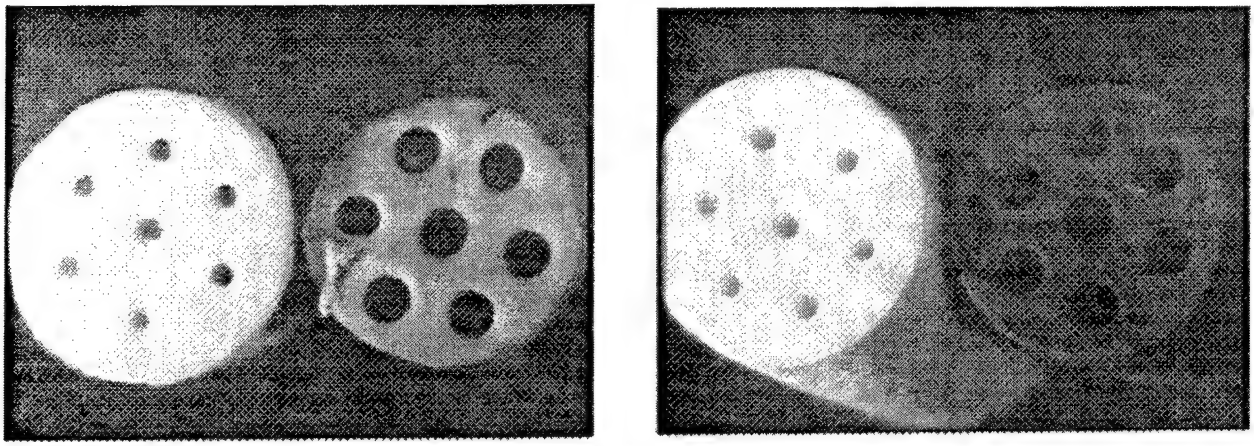
Originally, it was hoped that a propellant burning rate could be determined for each closed-bomb trial using the P-t curves and the changes in the grain dimensions of extinguished samples of plasma and conventionally ignited M30 and JA2 [3]. However, the fact that the P-t curves of the plasma-ignited grains show an initial rapid pressurization made the analysis more complicated. With the interrupted burning tests, the approach used was to consider identical propellants fired under similar plasma power conditions for which the pressure-time histories were comparable, with the exception that they were extinguished at two different pressures. (See Figure 7, ETC curves for M30 and JA2.) The recovered grains were measured to determine the extent of regression between the two blow-out pressures. During the plasma pulse, the pressure would typically rise to about 30 MPa for PE capillaries (JA2 samples) and about 20 MPa for Mylar capillaries (M30 samples); therefore, comparison of the calculated regression (using conventional burn-rate parameters over the experimental pressure interval) with the measured propellant regression between two pressures beyond 30 MPa yielded the extent of the intrinsic postplasma burning-rate augmentation.

For either M30 or JA2, no evidence was found of increased burning rate after the plasma event (i.e., the measured grain regression is consistent with those predicted using conventionally measured burning rates for these propellants). For both M30 and JA2, grain regression at 35 MPa was consistently slightly greater than predicted, based on conventional burning rates, suggesting that there is burning-rate augmentation during the plasma event. Thus, it appears that the post plasma burn rates are not intrinsic. Details of this analysis are provided in Birk et al. [7].

Burning occurred to a greater extent in the samples ignited with black powder than with the plasma due to the fact that, with plasma-ignited samples, a significant fraction of the total pressure was due to plasma injection rather than propellant burning. This initial plasma pressurization occurs rapidly, leaving a shorter effective time for propellant burning to occur, compared to conventional ignition (see Figure 7). This occurred whether Mylar or PE capillaries were used in the plasma ignition. This is reflected by the extent of grain regression. For example, for M30 grains recovered at nominally 35 and 60 MPa, the grain regression measured for plasma-ignited samples (Mylar capillaries) was 0.23 and 0.33 mm, while for conventionally ignited samples, the regression was greater at 0.37 and 0.62 mm, respectively.

Another indication that burning had progressed to a greater extent in the conventional samples was apparent with the color change of the white virgin M30 grains to a golden brown in the extinguished samples (Figure 8[a]). Most recovered grains from the plasma ignition were blackened (Figure 8[b]). However, on surfaces where the propellants were shielded from the direct blast of the plasma, the M30 grains from plasma-ignited samples (100 MPa) appear a lighter cream than for those from conventionally ignited samples (75 MPa), which are a golden-brown color. This suggests a difference in chemical mechanism between the conventional and plasma ignition. These observations of Mylar-based plasma-ignited samples are consistent with previously reported results for samples generated with PE-based plasma ignition, in which plasma-ignited samples (100 MPa) appear a lighter cream color than for those from conventionally ignited samples (62 MPa), which are a golden-brown color; the extent of burning was comparable for these samples also [8].

3.2 Grain Archaeology. In order to facilitate the interpretation of the morphological analyses provided by the SEM, the original positions and final locations of the propellant grains were tracked. In the discussion that follows, the top tier refers to the 15 grains closest to the blow-out disc and the bottom tier refers to the 15 grains closest to the igniter. The closed bomb is referred to as bomb, and chamber refers to the 240-liter chamber used to interrupt the burning.



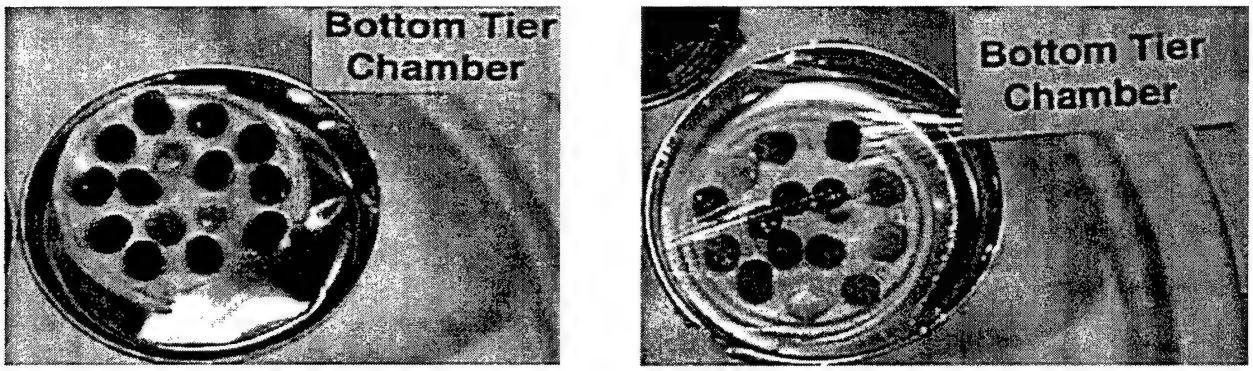
(a) (b)

Figure 8. Samples: (a) Conventionally Ignited With Black Powder and (b) Plasma-Ignited With Mylar-Based Capillaries.

More grains are recovered at 35 MPa blowout than at 60–70 MPa. At 35 MPa, grains remaining in the bomb were primarily from the bottom outer region; grains from the middle all passed through the bomb and entered the chamber and the foam, whether from the top or bottom tiers. At higher pressures, only the bottom outer grains are found in the bomb.

Visually, the grains recovered from the plasma-ignited samples appear differently than conventionally ignited samples. In the case of plasma-ignited samples, the top grains are light cream in color and the bottom grains are covered with a black residue. The residue is less dense for the Mylar-based capillary samples than for the PE-based capillary samples. At 35 MPa, the residue appears mainly on the grains originally located in the bottom tier nearest the igniter (Figure 9[a]).

Conventionally ignited samples (35 MPa) also exhibited some residue, presumably from the black powder ignition, but it is a less dense coating than for the plasma residue and is mainly limited to the bottom regions of the bottom middle grains (Figure 9[b]). The conventionally ignited samples (35 MPa) appeared more golden in color, as though they had undergone greater decomposition than the plasma-ignited samples. The top-tier grains were all found in either the



(a) (b)

Figure 9. Grains From the Bottom Tier, Extinguished at 35 MPa and Recovered in the Collection Chamber: (a) Plasma Ignition, Mylar Capillary and (b) Conventional Ignition.

chamber or the foam. Those found in the foam were virtually the same color as the virgin material, except for areas that had a light coating of black powder residue (Figure 10, left side). At 35 MPa, the number of grains remaining in the bomb are comparable for plasma-ignited and black powder-ignited samples and mainly originate from the bottom-tier middle section. For plasma-ignited samples, these grains have the greatest level of residue from the capillary.

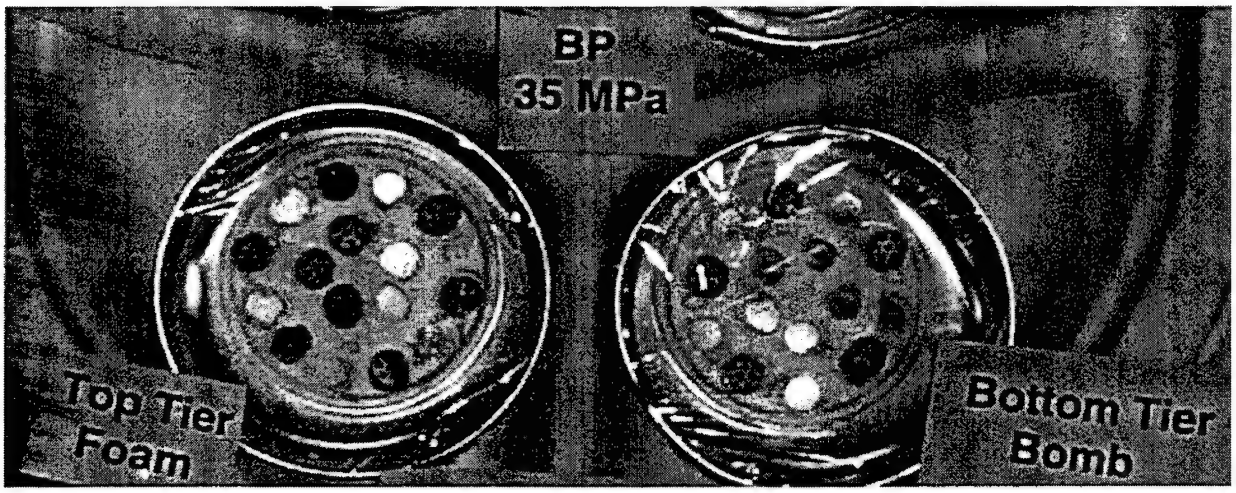


Figure 10. Grains Originally From the Top Tier, Extinguished at 35 MPa; Grains Recovered From the Closed Bomb (Right Side) Are Yellowed, While Grains Recovered in the Foam (Left Side) Are Not Discolored, Except for Modest Residue.

At 62–78 MPa, fewer grains (about 20 of the 30 originally packaged) are recovered for either conventionally or plasma-ignited samples. Most of the recovered grains were from the bottom tier and found in the chamber (not foam). The unrecovered grains primarily originated from the top tier; the top-tier grains that were recovered were mainly found in the foam. At this higher blow-out pressure, in most cases, no grains remain in the bomb for either ignition system. (Occasionally one remains.) For plasma-ignited samples at 70–78 MPa, most of the top-tier outer-ring samples are driven into the foam. In general, most samples embedded in the foam are lighter in color (i.e., not as golden brown) than those found elsewhere in the chamber. In contrast, very few bottom-tier samples are found in the foam, and they tend to be blackened from the plasma residue.

For 100-MPa blow-out and ambient temperature, only plasma-ignited trials were performed. There is little recovery of the top tier of grains, or any of the middle samples (top or bottom tier). Only bottom outer grains are found (9 of 10 original), and these were found in the chamber. No samples remained in the bomb at 100 MPa also. The plasma-ignited bottom outer grains at 100 MPa appear similar to the analogous grains from black powder ignition at 70 MPa. The extent of regression of grains from black powder ignition at 62 MPa was more comparable to those from plasma-ignited samples at 100 MPa than for plasma-ignited samples at 72 MPa.

It was desirable to investigate whether there is uniform burning/ignition of the samples with respect to location. Trying to do this based on the level of grain regression is complicated by the fact that the samples are located in different positions afterward (bomb, foam, chamber) and may not all extinguish simultaneously. Also, especially for plasma-ignited samples, the residue thickness appears to be significant relative to the changes in dimensions of the grains. The virgin M30 grains are somewhat irregular and do not have a smooth surface, increasing the standard deviation of the original grain measurements.

It is noted that for previous experiments [12] in which extinguished grains of JA2 were generated with plasma ignition (albeit at higher pressure—about 200–280 MPa), it was possible

to visually assess if sample fracture occurred during ignition or after being extinguished. This is because samples that fractured as a result of being extinguished had surfaces that were not blackened, since no burning occurred after fracture. Samples that fractured during ignition burned for a relatively long time and were blackened and discolored. However, in the current closed-bomb configuration, the results are not so simple to assess. Pressures are much lower, and the color change due to burning of the plasma samples is minimal; so, significant differences cannot be detected visually. Most surfaces are blackened by the plasma (often inside the perforations as well), so it is difficult to differentiate with the unaided eye between fracture due to the ignition-vs.-capture events. The SEM is very important in these assessments, and the reason for tracking the grains is for the purpose of correlating the SEM observations (e.g., unburned surfaces, particle embedding, etc.) to the proximity of the igniter.

The plasticizer results are consistent with previous results [3] using desorption - gas chromatography - mass spectroscopy (D-GC-MS), but the standard deviation for the high-performance liquid chromatography (HPLC) analysis is much lower. The D-GC-MS method had been used successfully in two previous studies to measure plasticizer depletion; but, in each case, significant changes in at least one of the components (DEGDN) was observed. In both of the prior cases, subsurface reaction or changes to the bulk of the propellant occurred. In one case, components migrated from a plasticizer-rich layer to another layer with no plasticizer [10]; in the second case, subsurface reaction and out-gasing were observed [8]. It was difficult to detect plasticizer depletion in the extinguished samples at low pressure, presumably because burning occurs just at the surface, and the overall bulk composition remains largely unchanged. Thus, for the Mylar-based experiments, plasticizer depletion at the surface was monitored by sampling only the outer propellant layer and was performed shortly after the propellants were extinguished to avoid plasticizer migration. Nonetheless, no component depletion or decomposition was detected.

3.3 LC-MS. The extinguished samples of JA2 and M30 at various blow-out pressures were analyzed; virgin samples were included as a reference. Only the outer surface of the grains was sampled in order to obtain the greatest possible concentration of any decomposition product

present. The LC-MS ion chromatograms obtained for JA2 and M30 extracts are shown in Figures 11 and 12, respectively. Chromatograms for the virgin and conventional JA2 samples are shown overlaid and are virtually indistinguishable. For the plasma-ignited sample, an early eluting peak (less than 1 min) is observed. Although the peak overlaps a methanol impurity peak (determined from a separate analysis of methanol; not shown), the peak is too large to be due to this component alone. Major peaks in the mass spectrum of this unknown were 63, 77, 120, and 148 m/z. It is noted that the nitrate esters (e.g., DEGDN and NG) typically yield a 46 m/z fragment, presumably due to ONO, and this fragment was not detected in the unknown. Thus, if it is a decomposition product of the NG or DEGDN, it would apparently be totally denitrated. The ion chromatograms of the M30 components (Figure 13) show that no decomposition product was detected.

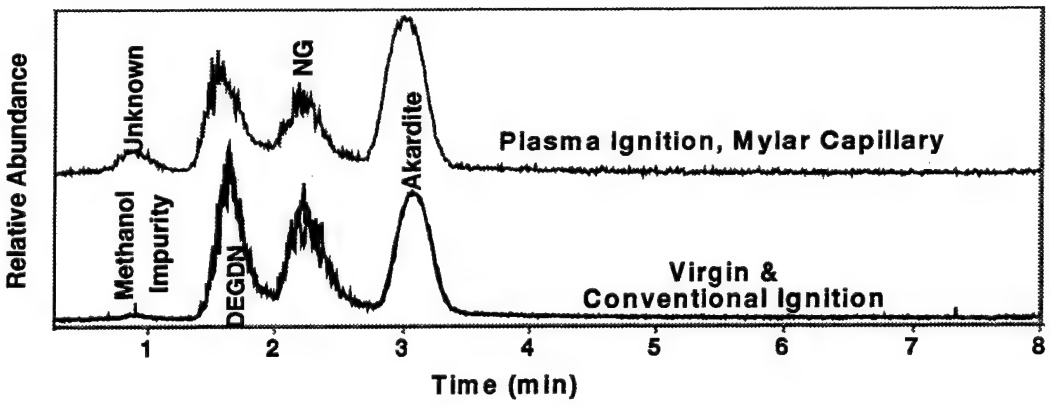


Figure 11. LC-MS Analysis of JA2 Samples.

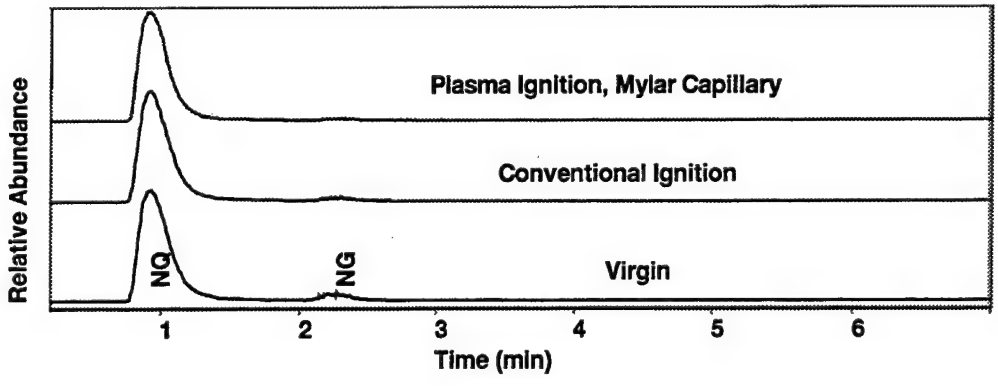
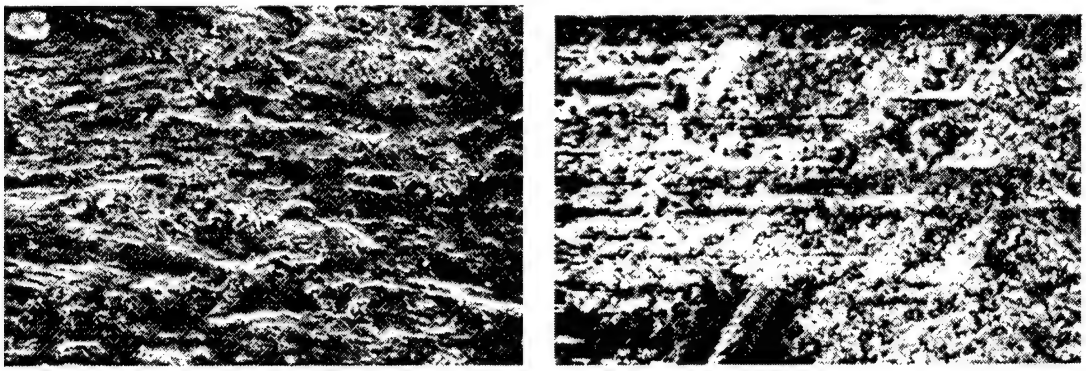


Figure 12. LC-MS Analysis of M30 Samples.

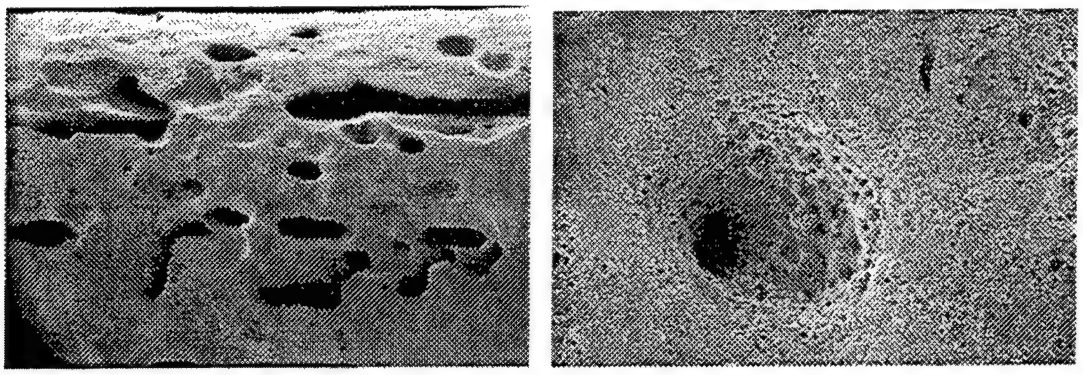


(a) Conventional Ignition (10 kpsi, 850×) (b) Plasma Ignition; PE Capillary (5 kpsi, 850×)

Figure 13. Micrograph of the Burning Surface of the Extinguished Grain Perforation Showing Different Features Between Ignition Methods.

3.4 SEM. Extinguished surfaces have been studied previously [12], and it has been noted that many combustion features are preserved on surfaces that undergo rapid pressure reduction. The flame is rapidly blown away from the surface, which quickly solidifies, leaving most of the burning surface features intact. SEM micrographs from this study revealed several interesting features. The exterior lateral surfaces of the extinguished grains from both the plasma and conventional ignition sources appeared very similar except for one feature. In plasma ignition, the grains showed evidence of burning caused by hot particles being sprayed onto the grain surface, as shown in Figure 14. This phenomenon would tend to augment the surface area available and give the appearance of a higher burning rate during the early portion of the propellant combustion. However, since the augmentation is caused by increased surface area, the apparent burning rate will be lowered in later stages of burning as the resulting surface area is reduced due to the intersection (burn through and crossing) of burning surfaces.

Another interesting feature was noted on the burning surfaces within the perforations. Most burning surfaces of extinguished propellant appear as shown in Figure 13(a), which depicts the conventionally ignited extinguished surface. Note that the surface is smooth, indicating melting. However, in Figure 13(b) there seems to be no indication of melting, and yet there appears to be a series of grooves that conform to the size of NQ particles, which appear at the same magnification in Figure 13(a). Stereoscopic pictures confirmed that groves were the features



(a) 5× (b) 90×

Figure 14. Micrograph of the Extinguished Grain Exterior Showing Evidence of Hot Particle Spray From the PE Plasma Igniter (100 MPa).

being observed. This suggests a different combustion process is in effect for the plasma-ignited samples that causes the NQ crystals to vacate the matrix. Note that, in Figure 13(b), the grooves are much less prominent at higher pressure (70 MPa or greater), and the surface appears more like the conventional. This is consistent with the burning mechanisms returning to similar processes at higher pressures.

One other observation on the morphology of the M30 propellant is worth noting. While most propellants have measurable melt layers that range from 2 to 30 μm in thickness, the melt layer for M30 is so thin that it was not able to be measured. The evidence for a melt layer is shown in Figure 13(a), as previously noted. However, when a cross section of that surface is made and the thickness measurement is attempted, no thickness can be determined because it appears to be so thin. The propellant goes from the outer surface to a structure identical to the unburned structure, regardless of the ignition source and without any measurable transition depth.

3.5 FTIR Analysis. Photoacoustic Spectroscopy (PAS) was previously used to investigate differences between conventionally ignited samples and those ignited with plasma using PE-based capillaries [8]. PAS successfully identified a trend for NQ to be diminished on outer surfaces (i.e., not perforations that were not examined) of the propellants. However, this effect was also

apparent using microreflectance spectroscopy. Additional bands were detected with the microreflectance technique, possibly because it is more surface specific than PAS. Thus, microreflectance was used to probe for decomposition products from the surface material sampled from the Mylar-based plasma-ignited experiments.

The previous microreflectance FTIR results [8] showed that JA2 ignited with either plasma or black powder showed evidence of aldehyde formation (appearance of an FTIR band at 1735 cm^{-1}), due to denitration of the nitrate esters. The microreflectance spectra of conventionally ignited M30 showed bands at 2885 cm^{-1} (aliphatic C-H) and 1100 cm^{-1}, which were less apparent in the PE-ignited samples. The conventionally ignited grains in this case were also a deep golden color; no grains of comparable color were found for the plasma-ignited samples.

In the recent work, a band appeared in the spectra of only M30 propellants, which were ignited with the plasma using Mylar capillaries. This band is also in the carbonyl region (1725 cm^{-1}) and appears at slightly lower frequency than the aldehyde band of JA2 (1735 cm^{-1}). Unlike the case of JA2 ignited with PE capillaries, however, the band in M30 did not appear in any of the conventionally ignited samples examined (Figure 15). The band was found (and was most intense) in all the spectra from samples extinguished at 35 MPa and in some of the samples up to 80 MPa; it was not found at the highest pressure used, 100 MPa, although an exhaustive profiling of the outer surfaces was not performed. It also has a different shape than the carbonyl bands normally observed by the authors in studying decomposition products of nitrate ester-based propellants.

The baseline for the extinguished samples in Figure 16 shows distortion that may be an artifact of sampling through the residue. This would render the sample "nonspecular," a criterion for using the Kramers-Kronig (KK) [13] transformation to obtain first the transmission and then the absorbance spectrum. Specular reflectance assumes a flat, nonscattering sample surface. It is also noted that the virgin M30 grains have a very irregular surface for which it is very difficult to achieve a useful reflectance spectrum. The spectrum for the virgin material achieved in Figure 17 was obtained by making a cross-sectional cut with a smooth surface.

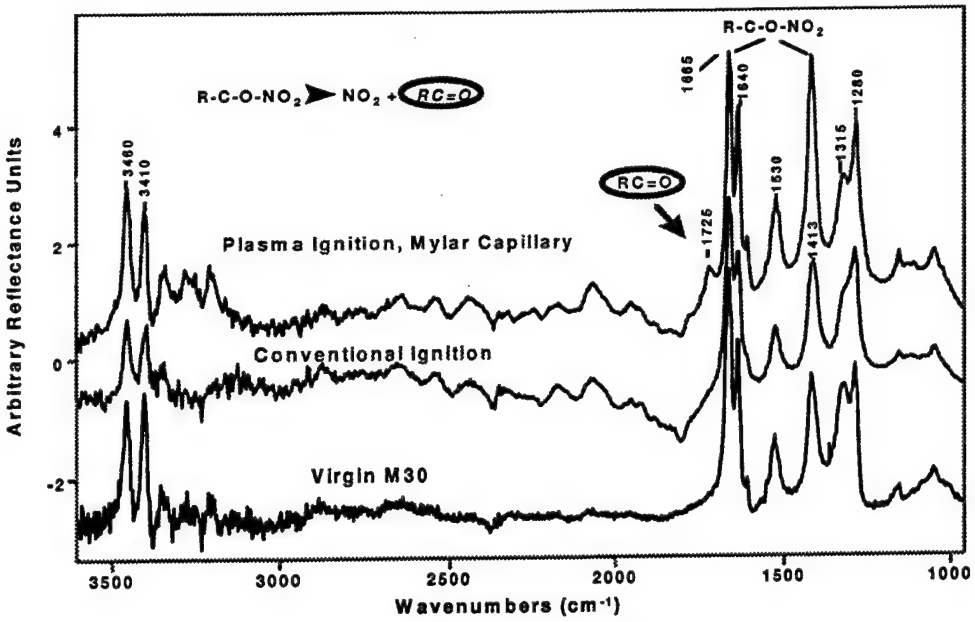


Figure 15. Spectra of Outer Surfaces of M30 Grains Exposed to Both Conventional and Plasma (Mylar Capillary) Ignition; the Spectrum of a Cross-Sectioned Sample of Virgin M30 Is Provided as Reference.

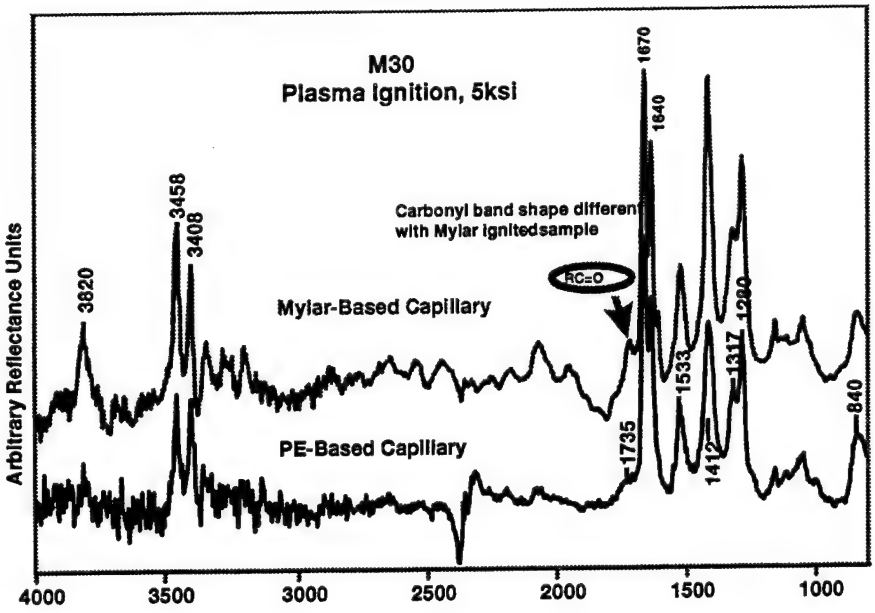


Figure 16. Spectra of Extinguished M30 Grains After Plasma Ignition With Mylar- and PE-Based Capillaries.

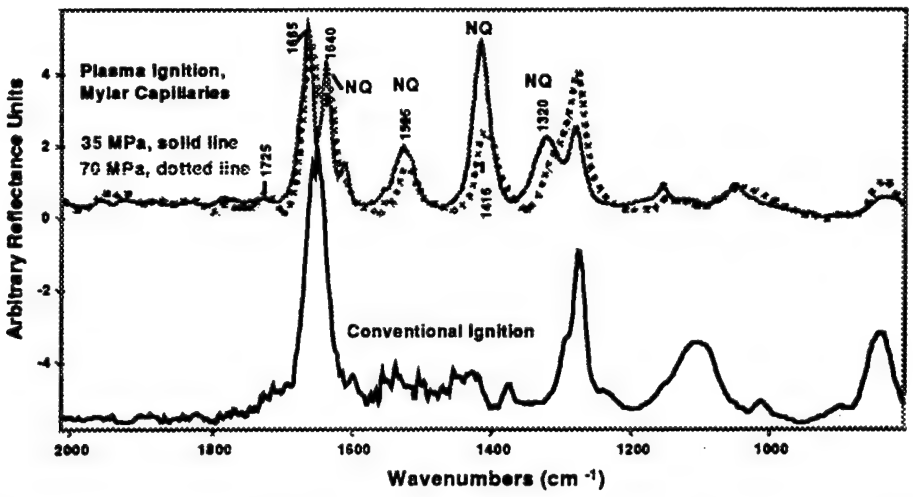


Figure 17. Spectra Showing NQ Diminishes With Increased Regression: Conventionally Ignited Samples Experience the Greatest Regression, and Plasma-Ignited Samples Extinguished at 35 MPa Experience the Least.

In previous work [8], the microreflectance spectra of M30 showed that the NQ level on the outer surfaces decreased with increasing extent of burning; this is true for conventionally and plasma-ignited (PE capillary) samples. This was also true for the perforation surfaces. This result can be resolved with that from the SEM results that showed that NQ particles were eliminated from the surface by the fact that the NQ crystals are locally concentrated and aligned along the direction of extrusion near the surface of the perforations. Microreflectance FTIR profiles about the top 10 μ, and, when NQ crystals are eliminated from the surface, the composition of the following sample is comparably rich in NQ crystals. Thus, the FTIR response is about the same whether or not the top NQ crystals are removed, whereas the SEM, with topological capability, detects the NQ crystal loss [8].

The current results with Mylar capillary plasma ignition also shows that the surface NQ level diminishes with increased regression; this was observed with conventionally and plasma-ignited samples. This is shown in Figure 17, consistent with the results obtained with PE-based capillaries discussed previously. Also evident in Figure 17 is a peak that appears in the spectra of conventionally ignited samples, centered at about 1100 cm^{-1}, which doe not appear distinctly in the spectra after plasma ignition from either PE- or Mylar-based capillaries. It is noted that

inorganic sulfur-containing compounds, as might be present with black powder ignition, have bands in the 1100-cm^{-1} region. However, the 1100-cm^{-1} band grew in relative intensity concomitantly with an aliphatic band (CH2, at 2975 cm^{-1}, not shown) as the pressure at which the samples were extinguished increased. Organic sulfur compounds generally absorb at frequencies higher than 1100 cm^{-1}. Thus, it is a possibility, but not likely, that the 2975-cm^{-1} and 1100-cm^{-1} bands are due to a sulfur compound.

3.6 XRF Spectroscopy. XRF was used to detect metals from plasma injection, which may impinge on the sample. The XRF results for the outer lateral surfaces of M30 were examined previously. Iron from the closed-bomb hardware was found on the outer surfaces for both plasma-ignited (PE capillary) and conventionally ignited samples. In addition, Cu from caps on the electrodes was deposited on the plasma-ignited samples. Since the SEM analysis showed differences in the grain perforation morphology between the plasma-ignited (PE capillary) and conventionally ignited samples, the perforations were also examined by XRF. The results are shown in Figure 18. As had been observed on the outer surface, the plasma-ignited sample shows the presence of Fe and Cu inside the perforations. However, although Fe was found on the outer surface of the conventionally ignited sample (Figure 18 insert), it is not observed inside the perforations. The spectrum of a virgin grain shows no significant Cu or Fe present.

The presence of metals inside the perforations in the case of plasma ignition, while none is found with black powder ignition, may suggest a greater force penetrates inside the perforations during plasma ignition. This could be consistent with the stripping of NQ (as evidenced with the SEM) observed with plasma ignition but not with conventional ignition. Analysis of plasma-ignited samples with Mylar capillaries is in progress. The results are very much of interest since the peak pressure generated in an empty closed bomb (i.e., no propellant) with Mylar (about 15 MPa) is less than with PE (over 30 MPa) [6]. It is possible that the Mylar capillary does not generate sufficient pressure to force material into the perforations.

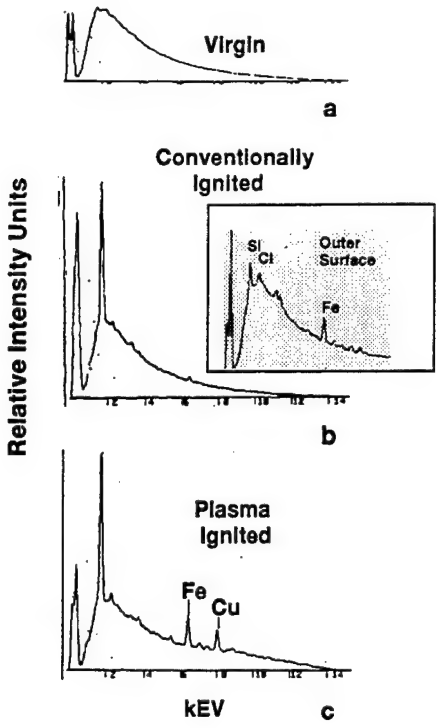


Figure 18. XRF Analysis of M30 Grain Perforations: (a) Virgin, (b) Ignited With Black Powder, and (c) Ignited With Plasma (PE Capillary).

4. Conclusion

The morphological and chemical characterization of extinguished M30 propellants from conventional and plasma (PE and Mylar) ignition has been performed. (The chemical characterization of M30 and JA2 samples from PE-based plasma ignition was, in part, presented previously [3]. Differences found between conventional and plasma ignition were primarily physical or morphological in nature. Extinguished grains burned to a greater extent than those ignited with plasma, due to the initial rapid pressure increase from the plasma pulse. This results in blow-out pressure being achieved more quickly for plasma ignition and in less time being needed for the propellants to burn. Thus, conventional samples extinguished at 35 MPa regressed about the same as plasma-ignited samples that burned to about 70 MPa. Plasma-ignited samples were largely covered with a residue from the capillary, whereas conventionally ignited samples only had traces of obvious residue, but the grains were not thickly coated. For the

plasma-extinguished grains, there are regions that are apparently sheltered from the residue. In these cases, the grains are less yellowed than grains that burned (i.e., regressed) to a similar extent. Also, the following were observed for the plasma-extinguished grains.

(1) There appears to be removal of NQ crystals in the perforations instead of the normal melt layer observed for conventionally ignited samples.

(2) Metals from ignition hardware are apparent in the perforations of the plasma-ignited samples (PE) but not for the conventionally ignited samples.

Some differences between PE and Mylar-based capillary plasma ignition were observed.

(1) The apparent burning-rate augmentation with Mylar is lower.

(2) The residue from the capillary is significantly less dense in the case of Mylar capillaries.

(3) The peak pressure measured in empty chamber firings is less for Mylar [3].

(4) Preliminary SEM results of samples from Mylar-based ignition have not shown evidence of removal of NQ from the perforations.

(5) Preliminary SEM results did not show evidence of hot embedded particles and increased surface area (SA) as observed with PE-based ignition.

Although extensive chemical characterization has been performed, there appears to be very little chemical difference between the burned surfaces and subsurfaces of the plasma-ignited and conventionally ignited samples. Efforts were undertaken to isolate products promptly after sampling. Although analyses of extracts of many grains by P-GC-MS and liquid chromatography - ultraviolet (LC-UV) were performed, no decomposition products or depletion

of propellant components were detected. Using LC-MS, a possible decomposition product from plasma-ignited samples was detected in the ion chromatogram; however, no identification from the mass spectrum has yet been possible. A slight decrease in NQ was detected by FTIR; however, this phenomenon is observed for both plasma and conventional ignition and has been reported for outer surfaces of other crystalline propellants under extinguished burn conditions as well [14]. Microreflectance FTIR did show that a species containing an aliphatic and 1100 cm^{-1} band was present in some conventionally ignited samples but not detected in any samples after plasma ignition; however this limited spectroscopic information is not sufficient to enable identification. Also, the position and shape of the carbonyl band that results from samples after Mylar-based plasma ignition is different than that observed previously from conventional or PE-based plasma ignition or any other treatment of nitrate esters studied by the authors. Whether or not this is due to different denitrated products being formed with the different treatments or is an artifact of sampling through the capillary residue (which is not feasible for the heavily coated samples from PE ignition) is being investigated. Nonetheless, it is not possible to assign specific differences in plasma-propellant interactions between conventional and plasma ignition from these limited observations.

In fact, it may be that there are few decomposition products remaining near the surfaces of these samples. SEM analysis revealed that, although a melt layer is present on the surface of M30, it is immeasurably thin. Thus, detecting products may be virtually impossible. Moreover, the SEM analysis showed that NQ depletion in the perforations was most evident at the lowest blow-out pressure used (35 MPa) and seemed to diminish at higher pressure, apparently becoming obscured by "normal" burning processes. Thus, plasma-propellant interactions, and associated effects, may occur only very early during ignition. This is consistent with the results reported by Birk et al. [7], that the increase in burning rate due to the plasma occurs during, not after the plasma event.

Birk has also analyzed the vivacity curves from the closed-bomb trials of the samples studied in this work. Vivacity curves [15], which slope upward with increasing pressure, indicate progressive burning, as would be expected from 7-perforated grains. Downward-sloping curves indicate regressive burning, which, if observed for 7-perforated grains, indicates an increase in

surface area due to some mechanism other than normal grain burning. Birk et al. [7] have found that with JA2, the grains appear to burn progressively, while, for M30, they appear to burn regressively. An increase in surface area with fractured grains is consistent with regressive burning.

The source of the increased surface is not certain at this time. However, the SEM photographs shown in Figure 13 reveal surface area generation not typical of normal burning of perforated grains. Kooker [16] has performed simulations showing that shape and magnitude of the burning-rate curves deduced from ETC closed-chamber experiments were reproduced with modest amount of grain fracture. It is feasible that an increase in surface area by other means could yield similar results. The level of surface area required to account for the apparent burning-rate augmentation and whether the phenomena observed in the SEM photographs could account for such surface area is under investigation. A depletion of NQ in the perforations will also be investigated as a means of contributing to a true burning-rate augmentation through chemical reactions. However, a true chemical burning-rate augmentation as the only mechanism for the M30 noninterrupted behavior would not be consistent with regressive burning.

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13. ABSTRACT *(Maximum 200 words)*

The morphological and chemical characterization of M30 propellants recovered after conventional and plasma ignition with polyethylene (PE) terephthalate (Mylar) in an interrupted closed bomb (extinguished at pressures between 35 and 100 Mpa) has been performed. It has been reported that burning rate augmentation appears to occur during the plasma event, but there was no evidence for post-plasma augmentation. For the extinguished grains from PE plasma ignition, there appears to be removal of nitroguanidine (NQ) crystals in the perforations. Also, hot embedded particles and increased surface area were observed, instead of the melt layer normally obtained with conventionally ignited samples. Although extensive chemical characterization has been performed, there appears to be very little chemical difference between the burned surfaces and subsurfaces of the plasma and conventionally ignited samples. Scanning electron microscopy (SEM) analysis revealed that although a melt layer is present on the surface of M30, it is immeasurably thin. Moreover, the SEM analysis showed that NQ depletion in the perforations was most evident at the lowest blow-out pressure used (35 Mpa) and seemed to diminish at higher pressure, apparently becoming obscured by "normal" burning processes. Thus, plasma-propellant interactions and associated effects may occur only very early during ignition, and it may be that there are few decomposition products remaining near the surfaces of the samples.

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7. If indicating a Change of Address or Address Correction, please provide the Current or Correct address above and the Old or Incorrect address below.

OLD ADDRESS

_____
Organization

_____
Name

_____
Street or P.O. Box No.

_____
City, State, Zip Code

(Remove this sheet, fold as indicated, tape closed, and mail.)
(DO NOT STAPLE)